

Speciation of Contaminants in Soil at the micron level

G.M.Lamble¹, R.J.Reeder², A.A.MacDowell³, R.S.Celestre³, H.A.Padmore³

1. Earth Sciences Division, Lawrence Berkeley National Lab., Berkeley Ca 94720

2. SUNY Stony Brook, NY 11973

3. Advanced Light Source, Lawrence Berkeley National Lab., Berkeley Ca 94720

INTRODUCTION

The complex heterogeneity of soils has precluded any direct measurements of contaminant speciation. The most reliable of speciation measurements to date have been made indirectly, for instance by chemical extraction methods (ie. by applying a series of chemical reagents to infer how the contaminant is bound within the many mineral and organic components within the soil). These indirect methods generally cause alterations to the true soil-contaminant system in the process of trying to make the measurements and are thus notoriously inaccurate.

EXPERIMENTAL

The newly developed micro X-ray absorption spectroscopy (micro-XAS) facility at the ALS provides a direct spectroscopic probe for contaminants in soils in their natural state. Here we apply a combination of two techniques: μ X-ray Fluorescence and μ XAFS. The μ X-ray Fluorescence measurements determine elemental distribution by irradiating the sample with a micron sized beam of white light or light of a fixed energy, then rastering the sample position to obtain an elemental distribution map over a chosen area. The μ XAFS measurements involves energy scanning of a monochromatic micro-beam on a chosen location, to determine the elemental speciation. The critical factors which make this possible are the high brightness of the ALS and advanced X-ray optics which provide small beams with sufficient intensity for making meaningful spectroscopic measurements on the same scale as the so-called "reactive-fraction" of soil minerals (i.e., < 2 microns).

Figure 1 shows a demonstration of these two types of measurement. The optical micrograph shows the area of interest within a sample of Long Island soil particles contaminated by Cr and Pb. The area sampled is 80 X 60 microns. Three elemental maps of Fe, Cr and Pb are shown as determined by X-ray fluorescence. Fe was measured to get an indication of any correlations in the metal distributions as Fe and Mn oxides are often considered influential in the binding of heavy metals. As can be seen from the elemental maps, the contaminants are highly localized (in regions of a few microns in size). The micro-XAS measurements were made by placing the monochromatic micro-beam in these localized regions of greatest contaminant concentration and then scanning over a characteristic absorption edge. Significantly, in the case of Cr we can observe a pre-edge feature which is characteristic of some presence of Cr(VI), which is the oxidation state of greatest concern in terms of its mobility and toxicity.

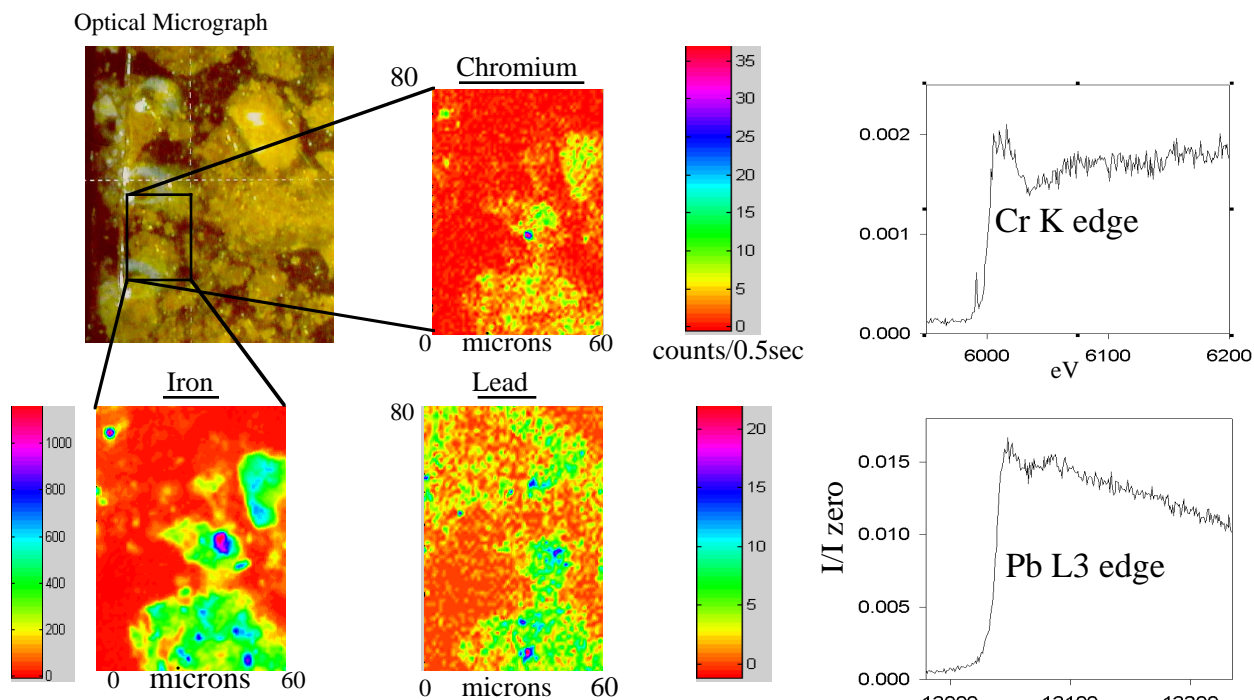


Figure 1. Optical micrograph (top left) of contaminated soil particles and associated elemental maps of Cr, Fe and Pb. The Cr and Pb near edge XAS are also included.

Speciation using unaltered samples of contaminants in such heterogeneous environments as soil represents one of the most important challenges for environmental analysis. When provided in combination with μ X-ray Fluorescence for elemental analysis we can additionally monitor the elemental associations of species with particular minerals. Along with knowledge of the species, this determination of the mineral association is also of central importance for devising effective remediation strategies.

REFERENCES

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Principle Investigator: Geraldine Lamble, Earth and Space Science Division, Lawrence Berkeley National Lab., Berkeley Ca 94720. Email: GMLamble@lbl.gov. Tel:510 495 2442